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COLUMBIUM ALLOY EXTRUSION PROGRAM

PHASE V: TUBING PROGRAM  
INTERIM REPORT VIII  
15 JANUARY 1963 - 15 APRIL 1963

BASIC INDUSTRIES BRANCH  
METALLURGICAL PROCESSING BRANCH  
AERONAUTICAL SYSTEMS DIVISION  
AIR FORCE SYSTEMS COMMAND  
UNITED STATES AIR FORCE  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

ASD PROJECT NO. 7-775

The final tube blank extrusion campaign is described, and the tube reducing program for extruded tube blanks of B-66 (Cb-5Mo-5V-1Zr) and D-43, previously designated X-110, (Cb-10W-1Zr-0.1C) is discussed. The processing is nearing completion on part of the D-43 tubes. The cracking of the B-66 during the first tube reduction has persisted.

(Prepared under Contract AF33(600)-40700 by E. I. du Pont de Nemours and Company, Inc., Baltimore, Maryland, E. V. Peterson)

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## FOREWORD

This Interim Technical Documentary Progress Report covers the work performed under Contract AF33(600)-40700 from 15 January 1963 to 15 April 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with E. I. du Pont de Nemours & Company, Inc., Baltimore, Maryland was initiated under Manufacturing Methods Project 7-775, "Columbium and Columbium Alloy Extrusion Program". It is being accomplished under the technical direction of Mr. T. S. Felker of the Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Mr. E. V. Peterson, Development Engineer, Metals Center, Baltimore, is the engineer directly responsible for the work. Others who cooperated in the development program were: Mr. J. S. Clark, Technical Supervisor, and Mr. J. A. Crane, Laboratory Engineer.

Wolverine Tube Division of Calumet Hecla, Inc., is the subcontractor to E. I. du Pont de Nemours & Company, Inc., for the tube reduction development program. Mr. J. C. Huber is the engineer directly responsible for the work at Wolverine. Mr. F. C. Eddens, Manager, Special Metals Department, and Mr. L. B. Moorman, Project Metallurgist, also contributed to this program.

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

## PUBLICATION REVIEW

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INTERIM TECHNICAL PROGRESS REPORT NO. 7-775 (VIII)  
15 JANUARY 1963 - 15 APRIL 1963

COLUMBIUM AND COLUMBIUM ALLOY EXTRUSION PROGRAM  
TUBING PROGRAM

ABSTRACT

The extrusion of three B-66 (Cb-5Mo-5V-1Zr) billets completed the extrusion part of the program.

The processing of D-43, previously designated X-110, (Cb-10W-1Zr-0.1C) tube blanks was continued in the third three-month period of Phase V. Some of the blanks have been processed through four reductions with 2600°F anneals after the first and third reduction. The outside surfaces are good. The inside surfaces have been improved by in-process conditioning, but a few defects still remain.

Attempts to make first reductions on the B-66 (Cb-5Mo-5V-1Zr) blanks resulted in cracking. The conditions that appear necessary for tube reducing the B-66, namely warm working, are beyond the scope of the present contract. Consequently, the work with B-66 was suspended, except for one piece of material which survived the first tube reduction.

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## I. INTRODUCTION

This report summarizes the results of the work performed during the third three-month period of Phase V of the "Columbium Alloy Extrusion Program". The first two three-month periods of Phase V have been summarized in (1) and (2)<sup>1</sup>.

The goal of Phase V is to produce three sizes of tubing of the columbium alloys B-66 and D-43 as follows:

1/2" O.D. x 0.062" wall (40% by weight)

3/8" O.D. x 0.062" wall (40% by weight)

1/4" O.D. x 0.018" wall (20% by weight)

The processing steps are -

- 1) double vacuum arc melt 8" diameter ingots
- 2) extrude to 4" rounds
- 3) machine hollow billets and re-extrude to  
1.750" O.D. x 0.30" and 0.25" wall tube  
blanks
- 4) tube reduce to finished tubing

Wolverine Tube Company, Allen Park, Michigan, is subcontractor to Du Pont for the tube reduction program. Wolverine and Du Pont have engaged in a cooperative program to determine a satisfactory reduction-anneal schedule for B-66 and D-43 tubes.

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<sup>1</sup> Numbers in parentheses refer to the Bibliography at the end of the report.



## II. SUMMARY OF PROGRESS

The final three B-66 tube blank extrusions in this program were performed during the reporting period. Two tube blanks with fair to poor surfaces were obtained. One extrusion stalled the press. The tube blanks were successfully straightened at a temperature of 500°F in an hydraulic press.

Continued efforts to make the first reduction on B-66 tube blanks resulted in cracking. However, one blank survived the first pass with one major radial crack. After a meeting with Westinghouse and Wolverine Tube personnel, it was concluded that the conditions desired for processing of B-66, namely working at approximately 500°F, were beyond the scope of work covered by the present contract. Funds were not available on this contract to pursue the warm working. Work on B-66 was suspended, except for the one piece which took the first reduction. This piece was annealed at 2600°F after the first reduction and subsequently given the second reduction to 1.062" O.D. x 0.147" wall. No additional cracking occurred, but the I.D. surface was extensively pitted.

The processing of D-43 has progressed satisfactorily with the major problem being internal conditioning to remove surface defects. The general surface condition of the I.D. has been improved by additional reductions and conditioning between reductions, but a few defects still remain.

Six D-43 tube blanks have had two reductions (to 1.062" O.D. x .100" and .147" wall), four blanks have had three reduc-

tions (to .815" O.D. x .065" wall), and two blanks have had four reductions (to 0.625" O.D. x 0.073" wall). Anneals at 2600°F were given after the first and third reductions, and the blanks were extensively conditioned on the I.D. after the first, second, and third reductions.

Minor end splitting on some blanks was cropped before further reduction.

### III. TECHNICAL DATA

#### A. TUBE BLANK EXTRUSIONS

Three B-66 billets which had stalled in the third series of extrusions, as indicated in Interim Report VII, were remachined. An attempt was made to salvage this material by extruding according to the best extrusion practices as described in Interim Report VI and VII. The dies and mandrels were the same as described in Interim Report VII. Briefly, the dies were the 90° cone type. The working surfaces of both the dies and mandrels were zirconium-oxide coated. The finished coatings, after grinding, were approximately 0.030" thick. The extrusion size would allow approximately 0.050" of material for "clean-up" on both the inside and outside surfaces of the tube blank (tube blank size was 1.750" O.D. x 0.300" wall). The billets were machined as shown in Figure 1.

The extrusion procedure was as follows:

1. Heat billet in induction heater under protective atmosphere, at rate of 150-200°F/minute. Measure temperature by optical pyrometer. Soak 5 minutes.
2. Lubricate liner with mixture of MoS<sub>2</sub> and "Oildag".<sup>1</sup>
3. Insert die and mandrel (preheated in 750°F oven for 1/2 hour) in press.
4. Transfer billet from heater to press in less than 35 seconds. Roll heated billet over powdered glass on glassing table in transfer; apply powdered glass to bore manually.

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<sup>1</sup> Proprietary lubricant manufactured by Acheson Colloids Company.

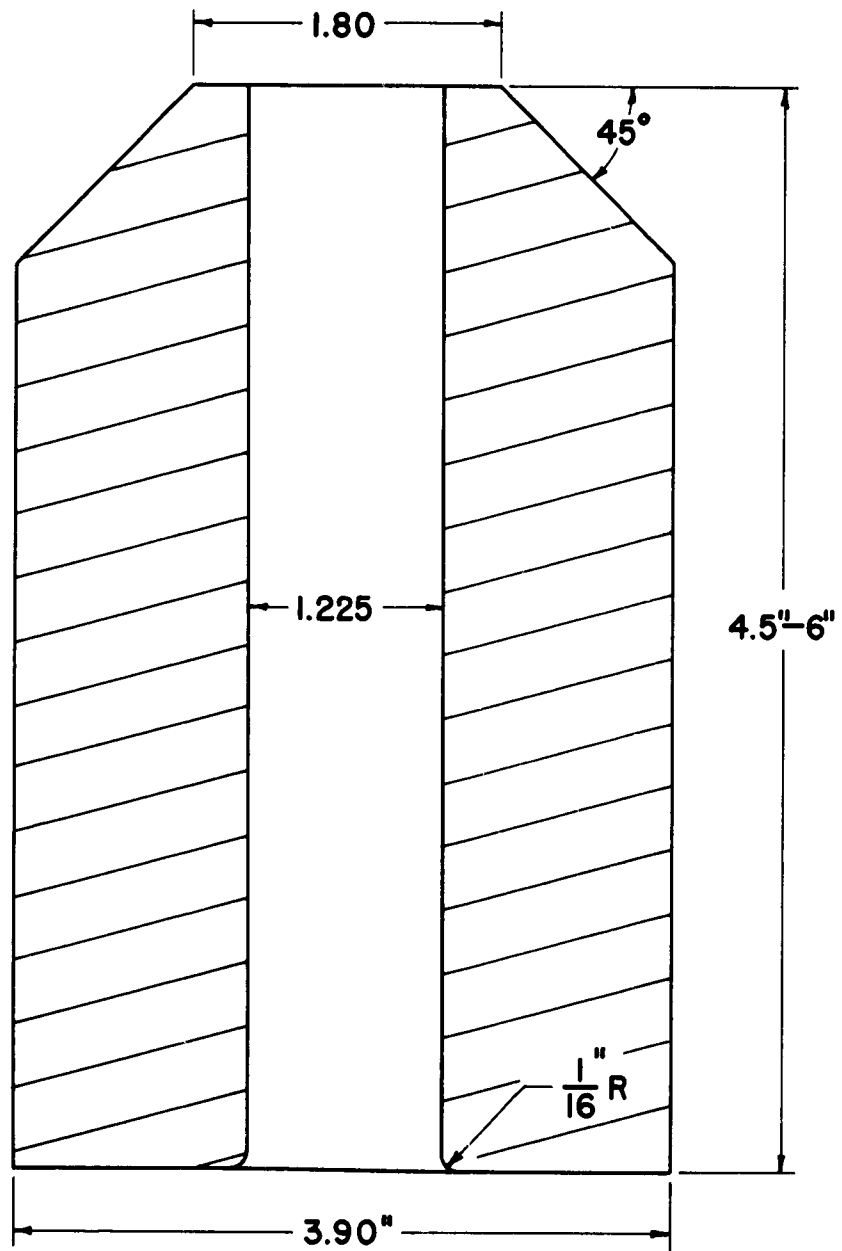


FIGURE 1

BILLET DESIGN FOR TUBE BLANK EXTRUSIONS

5. Place two 1" thick graphite pads heated to 2200°F directly behind billet. Place a 2" thick copper follower block behind graphite.

6. Extrude at ram speed of approximately 5"/second.

The results of the extrusions are shown in Table 1. One extrusion stalled the press. One of the other two extrusions produced a fairly good tube blank, but the other one was too rough on both I.D. and O.D. surfaces to warrant further processing.

TABLE 1  
B-66 TUBE BLANK EXTRUSIONS  
(in order of performance)

<u>Billet</u>	<u>Die Type</u>	<u>Extrusion Ratio</u>	<u>Billet Temp Of</u>	<u>Extrusion Pressure, ksi</u> <u>B.T.</u>	<u>Running</u>	<u>Results</u>
B-66-277-03	Cone	7.5:1	2940	190	--	Stalled
B-66-279-04	Cone	7.5:1	3100	190	144-170	O.D. surfaces fair I.D. surfaces fair
B-66-347-02	Cone	7.5:1	3080	190	143-160	O.D. surfaces poor I.D. surfaces good over first half of extrusion, and poor over last half

## B. STRAIGHTENING TUBE BLANKS

Previous attempts to straighten B-66 and D-43 tube blanks, (room temperature), in both a roller straightener and in a gag press have resulted in cracking of the blanks<sup>(2)</sup>. Several blanks of both alloys, as-extruded and deep-pickled, were successfully straightened in a gag press after preheating the blanks for approximately 1/2 hour in an oven at 550°F. Each blank was taken immediately to the press from the oven and worked during an approximate one-minute period. No breaking occurred and no cracking was evident. The degree of straightness obtained in the gag press was variable and not accomplished with the degree of precision associated with roller straightening. However, this operation demonstrated that tube blanks of both alloys, when given the preheat treatment, could be straightened sufficiently for tube reduction.

### C. TUBE REDUCTION

Wolverine Tube, Allen Park, Michigan, is the subcontractor who is performing the tube reductions in a cooperative program with Du Pont. The process schedules, set up by Wolverine and agreed to by Du Pont, are shown below as Schedule No. 1 (1/2" and 3/8" O.D. tubing) and Schedule No. 2 (1/4" O.D. tubing).

At the time Interim Report VII was issued, ten tube blanks had been put through the first reduction; seven of these were D-43 (X-110), and three were B-66. All three B-66 blanks cracked radially, two of them after careful hand conditioning and repickling. The D-43 blanks were reduced successfully. All blanks (D-43 and B-66) were in the annealed condition, and both materials were comparable with respect to surface condition and size and type of defects present<sup>(2)</sup>.

During the third quarter of Phase V, an additional B-66 blank, annealed and extensively conditioned to remove all visible defects, cracked during the first reduction. Three more B-66 blanks in the "as-extruded" condition were given the first reduction. Two of these failed to take the reduction. The third was reduced, with one large radial crack evident.

Because of the general lack of success in making the first tube reductions on B-66, work on B-66 was halted to permit study of the problem.

Room temperature bend tests were made on machined specimens of B-66 tube blanks in order to get an indication of room temperature ductility. The specimens were approximately 1/8" thick x 5/16" wide x 2" long, and were taken longitudinally



PROCESS SCHEDULE NO. 1

Tube Reduction to 1/2" and 3/8" O.D. Tubing

Finish target sizes: .500"  $\pm$  .005" O.D. x .060"  $\pm$  10% wall (40%)<sup>1</sup>

.375"  $\pm$  .005" O.D. x .060"  $\pm$  10% wall (40%)<sup>1</sup>

Starting material: 1.750" O.D. x .300" wall

	<u>Operation</u>	<u>Size</u>	<u>% Wall Reduction</u>	<u>% Area Reduction</u>
1.	Inspect	1.750" ODx.300" wall	-	-
2.	<u>Tube reduce</u>	1.375" ODx.210" wall	30	43.8
3.	Inspect			
4.	<u>Anneal</u>			
5.	Salvage; inspect			
6.	<u>Tube reduce</u>	1.062" ODx.147" wall	30	44.9
7.	Inspect			
8.	Salvage; inspect			
9.	<u>Tube reduce</u>	.812" ODx.103" wall	30	45.9
10.	Inspect			
11.	<u>Anneal</u>			
12.	Salvage; inspect			
13.	<u>Tube reduce</u>	.625" ODx.073" wall	30	45.0
14.	Inspect			
15.	Salvage; inspect			
16.	<u>Tube reduce</u>			
	half to .500" ODx.060" wall		30	34.1
	half to .375" ODx.060" wall		30	53.2
17.	Inspect			
18.	<u>Anneal</u>			
19.	Salvage			
20.	Final inspection			

<sup>1</sup> Percentage based on total material supplied by Du Pont

PROCESS SCHEDULE NO. 2

Tube Reduction to 1/4" O.D. Tubing

Finish target size: .250"  $\pm$  .005" O.D. x .015/.020" wall (20%)<sup>1</sup>

Starting material: 1.750" O.D. x .250" wall

	<u>Operation</u>	<u>Size</u>	<u>% Wall Reduction</u>	<u>% Area Reduction</u>
1.	Inspect	1.750" ODx.250" wall	-	-
2.	<u>Tube reduce</u>	1.375" ODx.160" wall	35	47.5
3.	Inspect			
4.	<u>Anneal</u>			
5.	Salvage; inspect			
6.	<u>Tube reduce</u>	1.062" ODx.100" wall	35	50.8
7.	Inspect			
8.	Salvage; inspect			
9.	<u>Tube reduce</u>	.812" ODx.065" wall	35	49.5
10.	Inspect			
11.	<u>Anneal</u>			
12.	Salvage; inspect			
13.	<u>Tube reduce</u>	.625" ODx.038" wall	42	53.9
14.	Inspect			
15.	Salvage; inspect			
16.	<u>Tube reduce</u>	.500" ODx.022" wall	42	52.8
17.	Inspect			
18.	<u>Anneal</u>			
19.	Salvage; inspect			
20.	<u>Tube reduce</u>	.250" ODx.018" wall	18	60.6
21.	Inspect			
22.	<u>Anneal</u>			
23.	Salvage			

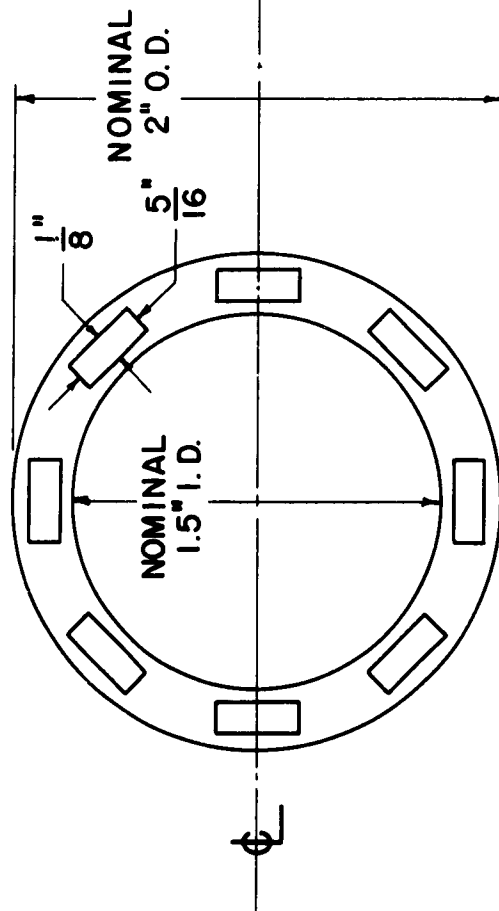
24. Final inspection

<sup>1</sup> Percentage based on total material supplied by Du Pont

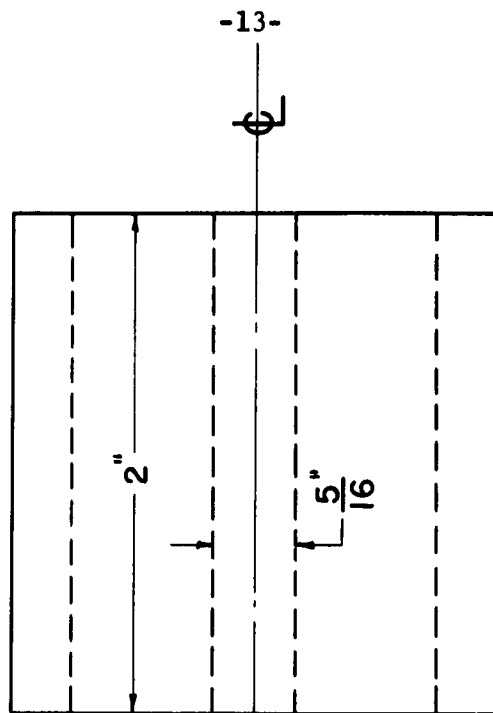
from the wall of the tube blank and centrally located within the wall thickness, Figure 2. The specimens were tested (1) as-extruded, (2) annealed at 2400°F for 1 hour, and (3) annealed at 2600°F for 1 hour. The tests were made without strain rate measurements on a slow acting hydraulic press. All specimens withstood a 90° 2T bend without any evidence of cracking. Further bending of the same specimens without physical restraint to a 180° and approximately 1/2T bend showed no cracking of any of the annealed specimens. Two of four "as-extruded" specimens broke just prior to completion of the 180° bend. This behavior indicated a high degree of ductility under slow bend conditions at room temperature.

A meeting was held with Westinghouse and Wolverine Tube to discuss the B-66 cracking problem. The results of the tube reducing attempts, the bend test results, and the Westinghouse experience with the alloy were discussed. It was concluded that the most favorable approach to the reduction of the B-66 tube blanks would be warm working (approximately 500°F). It was felt that the extruded B-66 was too strain rate sensitive at room temperature to readily permit making the first tube reduction under present conditions, and that its workability would be appreciably better at 500°F. Inasmuch as warm working is beyond the scope of the present contract, and in view of the small amount of usable B-66 tube blank stock remaining, and of the limited funds available, no further first reductions were made.

The one B-66 blank that did withstand the first reduction, with one large crack, was annealed at 2600°F, I.D.



END VIEW OF SAMPLE  
SHOWING LOCATION OF  
BEND SPECIMENS



SIDE VIEW OF SAMPLE

FIGURE 2

LOCATION OF BEND SPECIMENS IN TUBE BLANK SAMPLE

conditioned, and second reduced to 1.062" O.D. x 0.147" wall. No additional cracking occurred, but the I.D. surfaces were extensively pitted. A length of about 24", which is in good condition, will be processed.

The reduction of the D-43 tube blanks has proceeded according to the processing schedules referred to above. As many as four reductions, with in-process anneals at 2600°F after the first and third reductions, have been made on some of the tube blanks. Limited end splitting has occurred, but was readily controlled by cropping. The major problem continues to be conditioning of residual extrusion defects from the inside surfaces. The defects have been reduced in size and number by rather extensive conditioning between tube reductions, but some remain.

A summary of the tube reducing results of B-66 and D-43 is given in Tables 2, 3, and 4.

TABLE 2

SUMMARY OF RESULTS OF FIRST TUBE REDUCTION

<u>Identification</u>	<u>Base Tube Heat Treatment</u>	<u>Tube Reduced to Size</u>	<u>Tube Reducing Schedule No.</u>	<u>Tube Reducing Pass No.</u>	<u>Results</u>
D-43-278-02B	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	I.D. Defects
D-43-280-01B	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	I.D. Roughness. End splitting
D-43-280-07B	Annealed at 2600°F	1.375"O.D.x.160"W	2	1st	I.D. Defects
D-43-346-01B	Annealed at 2600°F	1.375"O.D.x.160"W	2	1st	I.D. Defects
D-43-346-03B	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	I.D. Roughness
D-43-278-03F	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Satisfactory
D-43-278-05	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Satisfactory
D-43-280-03F	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Satisfactory
D-43-280-04	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	I.D. Defects; some O.D. surface cracks
D-43-280-06F	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Satisfactory
D-43-280-06B	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	O.D. Surface good; some I.D. pits
B-66-277-01F	Annealed at 2600°F	1.375"O.D.x.160"W	2	1st	Tube cracked

D-43-346-01B	Annealed at 2600°F	1.375"O.D.x.160"W	2	1st	I.D. Defects
D-43-346-03B	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	I.D. Roughness
D-43-278-03F	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Satisfactory
D-43-278-05	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Satisfactory
D-43-280-03F	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Satisfactory
D-43-280-04	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	I.D. Defects; some O.D. sur- face cracks
D-43-280-06F	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Satisfactory
D-43-280-06B	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	O.D. Surface good; some I.D. pits
B-66-277-01F	Annealed at 2600°F	1.375"O.D.x.160"W	2	1st	Tube cracked
B-66-277-01B	Annealed at 2600°F	1.375"O.D.x.160"W	2	1st	Tube Cracked
B-66-279-01F	Annealed at 2600°F	1.375"O.D.x.210"W	1	1st	Tube broke in tube reducer
B-66-279-01B	Annealed at 2600°F and extensively conditioned	1.375"O.D.x.210"W	1	1st	Tube broke in tube reducer and I.D. gall- ing occurred
B-66-277-05B	As Extruded	1.375"O.D.x.210"W	1	1st	Tube broke in tube reducer
B-66-279-02F	As Extruded	1.375"O.D.x.210"W	1	1st	Good. I.D. pits; O.D. galling; one radial crack
B-66-347-04B	As Extruded	1.375"O.D.x.210"W	1	1st	Broke in tube reducer

TABLE 3

SUMMARY OF RESULTS OF SECOND TUBE REDUCTION

<u>Identification</u>	<u>Starting Condition</u>	<u>Tube Reduced to Size</u>	<u>Tube Reducing Schedule No.</u>	<u>Tube Reducing Pass No.</u>	<u>Results</u>
D-43-278-02B	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	Good. I.D. honing required
D-43-280-01B	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	Good. I.D. honing required
D-43-280-07B	Annealed at 2600°F	1.062"O.D.x0.100"W	2	2nd	Very good. Isolated defects
D-43-346-01B	Annealed at 2600°F	1.062"O.D.x0.100"W	2	2nd	Very good. Isolated defects
D-43-346-03B	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	Good. I.D. honing required
D-43-278-05	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	Good
D-43-280-04	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	I.D. honing required
D-43-280-06F	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	End cracks 1-1/8" long
D-43-280-06B	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	Good. Isolated defects
D-43-278-03F	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	Light O.D. pick-up. Isolated defects
D-43-280-03F	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	I.D. defects. Honing required
B-66-279-02F	Annealed at 2600°F	1.062"O.D.x0.147"W	1	2nd	Extensive I.D. defects over part of tube



TABLE 4

SUMMARY OF RESULTS OF THIRD TUBE REDUCTION

<u>Identification</u>	<u>Starting Condition</u>	<u>Tube Reduced to Size</u>	<u>Tube Reducing Schedule No.</u>	<u>Tube Reducing Pass No.</u>	<u>Results</u>
D-43-278-05	As Tube Reduced thru 2nd pass	.812"O.D.x0.103"W	1	3rd	Good
D-43-280-04	As Tube Reduced thru 2nd pass	.812"O.D.x0.103"W	1	3rd	I.D. pick-up. Honing required
D-43-280-06F	As Tube Reduced thru 2nd pass	.812"O.D.x0.103"W	1	3rd	End splits 2" long
D-43-280-06B	As Tube Reduced thru 2nd pass	.812"O.D.x0.103"W	1	3rd	End splits 1" long
D-43-278-03F	As Tube Reduced thru 2nd pass	.812"O.D.x0.103"W	1	3rd	End splits and I.D. pits. Honing required
D-43-280-03F	As Tube Reduced thru 2nd pass	.812"O.D.x0.103"W	1	3rd	Deep I.D. defects

TABLE 5  
SUMMARY OF RESULTS OF FOURTH TUBE REDUCTION

<u>Identification</u>	<u>Starting Condition</u>	<u>Tube Reduced to Size</u>	<u>Tube Reducing Schedule No.</u>	<u>Tube Reducing Pass No.</u>	<u>Results</u>
D-43-280-06F	Annealed at 2600°F	.625"O.D.x.073"W	1	4th	Very good
D-43-280-06B	Annealed at 2600°F	.625"O.D.x.073"W	1	4th	Very good. Slight I.D. pick-up at one end

#### IV. FUTURE PROGRAM

##### A. TUBE REDUCTION

The tube reduction of D-43 will be continued as indicated on the Process Schedules Nos. 1 and 2.

Group I, now in process in the fourth reduction, will be given the fifth reduction to final size prior to the final anneal.

Group II, now ready for the third reduction, will be given the third reduction, annealed, then given the fourth reduction. At this point it is planned to alter the original processing schedule on part of the material to determine the effect of reduction-anneal schedule on final mechanical properties. Three pieces of tubing, one intended for each of the three final sizes, namely 0.500" O.D. x 0.060" wall, 0.375" O.D. x 0.060" wall, and 0.250" O.D. x 0.018" wall, will be given an additional anneal after the fourth tube reduction. The three pieces will then continue as per the original schedule. The balance of the material will follow the original schedule without an anneal between the fourth and fifth tube reductions.

##### B. FINAL INSPECTION AND EVALUATION

The final product will be given dimensional and surface inspections. Laboratory evaluation will include chemical analysis, photomicrographs, flare tests, and room temperature and 2200°F tensile tests.

A final report will be issued upon completion of the program.

V. ACKNOWLEDGEMENTS

The author wishes to acknowledge the contributions to this program by J. S. Clark for technical supervision, R. W. Felber for extrusion operations supervision, and J. A. Crane for metallography and mechanical testing.

Wolverine Tube is conducting the tube reduction and the in-process conditioning and inspection portion of the program. The valuable contributions of personnel of that company are acknowledged, in particular those of J. C. Huber, the engineer in charge, F. C. Eddens, Manager, Special Metals Department, and L. B. Moorman, Project Metallurgist.

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